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Relationship Between Industrial Discharges and Contamination of Raw Water Resources by Perfluorinated Compounds: Part II: Case Study of a Fluorotelomer Polymer Manufacturing Plant

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Abstract In this study, the concentrations of 10 perfluorinated compounds (PFCs) were measured in effluents of a fluorotelomer polymer manufacturing plant and its wastewater treatment plant. A 50-fold increase between the two effluents mass flows was observed. The water quality of two drinking water treatment plants located downstream at 15 and 25 km from the manufacturing plant was examined. An increase of the sum of PFCs was observed between the river (30 ng/L) and an alluvial well (70 ng/L), and between the raw water (9 ng/L) and the outlet of a biological treatment (97 ng/L). These results indicate a possible degradation of fluorotelomers, occurring during wastewater treatment, sediment infiltration in the alluvial aquifer, and drinking water treatment.

Keywords Drinking water · Contamination · Perfluorinated compounds · Industrial activities

Perfluorinated compounds (PFCs) are a group of chemical substances that are characterized by a fully fluorinated carbon chain. They do not occur naturally in the environment and have been synthetically produced since the 1950s. They are used in many types of products such as fire-fighting foams and powders, process aids in fluoropolymer manufacturing plants, grease-repellent paper, water- and dirt-resistant textiles, polishes and waxes, and ant insecticides (Kissa 2001; Renner 2001). They have been detected in wild animal tissues, human sera, soils, surface water and groundwater, sludge and wastewater, precipitation, air, and indoor dust. Some of them are persistent, bioaccumulative, and potentially hazardous substances.

Fluorotelomers (FTs) are a subgroup of PFCs which are partially fluorinated. A small carbon hydrogen chain (generally two carbons) links the perfluorinated carbon chain to a functional group, e.g. alcohol [FTOH: CF_3–$(CF_2)_n$–$(CH_2)_p$–OH], olefin (FTO), sulfonate [FTS: CF_3–$(CF_2)_n$–$(CH_2)_p$–SO_3H], iodide [FTI: CF_3–$(CF_2)_n$–$(CH_2)_p$–I)] or acrylate (FTA: CF_3–$(CF_2)_n$–$(CH_2)_p$–O–CO–CH=CH_2). FTs are commonly obtained by an industrial process called telomerization (Kissa 2001). These compounds are subsequently used in the synthesis of various fluorosurfactants or incorporated as building blocks in the production of fluorotelomer polymers (FTPs). FTPs are characterized by a hydrocarbon backbone and used for various applications including paints, carpets, and repellent paper coatings (Kissa 2001). These commercial and industrial products contain unbound residual FTs because the processes used to synthesize them do not always incorporate all of the fluorotelomers (Dinglasan-Panlilio and Mabury 2006).

Due to their carbon hydrogen chain, FTs can undergo degradation in the environment. The perfluorinated tail is stable and it is often this chemical chain that is detected in samples by analysts, as end-stage metabolites including perfluoroalkyl carboxylates (PFCAs) and perfluoroalkyl sulfonates (PFASs). Moreover, several authors have proposed degradation pathways and attempted to identify intermediate degradation products (Ellis et al. 2004; Liu et al. 2007; Myers and Mabury 2010). FTPs may also be subject to degradation and therefore a source of PFCs in the environment (Russell et al. 2010; Washington et al. 2009).

The goal of this field study was to estimate the impact of the effluents of a fluorotelomer polymer manufacturing plant to the water resources located downstream from the

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effluent discharge point. In this study, we targeted 10 PFCs, identified as end-stage metabolites of the compounds used and produced in this facility.

Materials and Methods

The fluorotelomer polymer production plant is located within an industrial park of about 500,000 m^2. Fire-fighting foams and stain repellents for paper, carpets and textiles have been produced by this manufacturer since it opened in 1996. Prior to 1996, another manufacturer occupied the plant, and perhaps also used or produced PFCs. Other facilities located in this industrial park produce ionic polymers, urea-formalin resins, coating resins for wood protection, and acrylic polymers. A specific wastewater treatment plant (WWTP) receives the wastewater generated by these industrial activities. Briefly, raw effluent enters in a primary treatment tank where pH is neutralized. Wastewater then flows to an activated sludge aeration basin. After this treatment, wastewater passes into a floatation tank before being discharged into the river (average river flow: 110 m^3/s). The digested sludge is sent for incineration.

In July 2010, water samples were collected at nine sampling sites from the industrial park. Selected sampling points included three monitoring wells used to collect water from the alluvial aquifer (hereafter referred as P01A, P02A, and P03A), two monitoring wells used to collect water from a deeper aquifer (hereafter referred as P01D, and P02D), raw sewage generated by the FTP plant (hereafter referred as WI), the end-stage effluent of the industrial WWTP (hereafter referred as WE), and the effluents of two basins that drain all the run-off waters of the industrial site (hereafter referred as NB and SB, respectively). The NB basin receives the run-off water from the north of the industrial area, and the SB basin from the south. According to the manufacturer, the facility is located in an area where all the run-off waters are received by the SB basin.

The effluent samples (WI and WE) were 24 h and 7 days flow-proportional, composite samples, whereas other samples were extemporaneously collected. The groundwater flows approximately from monitoring well P02A to P03A. P02A and P02D are the monitoring wells nearest to the FTP plant. In accordance with good sampling practices, the volume of each monitoring well was renewed twice before collecting the sample. Figure 1 is a map of the industrial site, which includes the FTP plant.

During the same sampling period, six raw water resources were also collected from two drinking water treatment plants (DWTP). The first DWTP was located 15 km downstream from the FTP plant. Raw water of the first DWTP (named DWA) is supplied by five alluvial wells (hereafter referred as DWA-1, DWA-2, DWA-3, DWA-4 and DWA-5). The plant uses sand filtration and chlorination to treat water. The drinking water produced by this plant is consumed by 80,000 people. The second DWTP was located 25 km downstream from the FTP plant. Raw water of the second DWTP (named DWB) is directly pumped into the river. Two treatment processes are used in parallel: a membrane filtration process and a biological treatment. Membrane filtration contributes to 80 % of the drinking water flow. The biological treatment technique includes settling, sand filtration, ozonation, activated carbon filtration and chlorination. The water produced by this plant is consumed by 650,000 people.

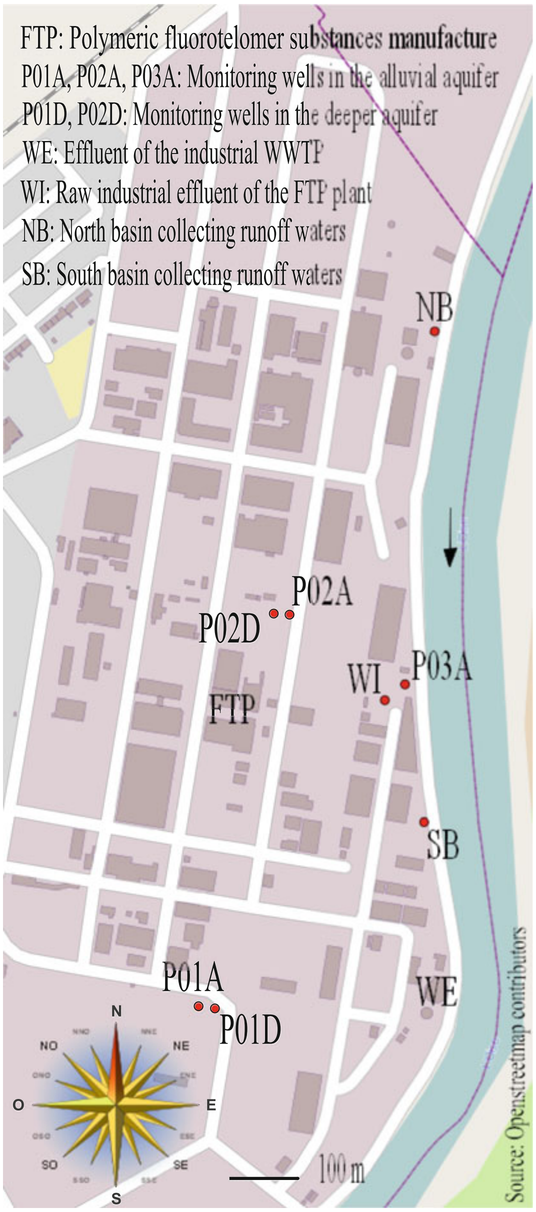

Fig. 1 A map showing sampling locations within the FTP plant

River water samples were also collected from three points during the same period. The sampling was performed from the river bank, approximately 15–30 cm below the surface of the water. The first sample (hereafter referred as RW1) was taken from the river at 500 m upstream from the FTP plant. The second sample (hereafter referred as RW2) was taken from the river at 500 m downstream from the point where the effluent of the industrial WWTP joins the river. The third sample (hereafter referred as RW3) was taken from the river at the foot of the DWA. The accurate location of the study area has been deliberately anonymized.

Samples were collected by the manager of the WWTP and members of a control laboratory. New 1 L high-density polyethylene bottles supplied by our laboratory were used to collect water samples. All water samples were stored at 5°C in the dark until extraction. To minimize the possibility of inadvertently introducing PFCs into samples, fluoropolymer materials were avoided throughout the experiment.

Ten PFCs were analyzed: perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), perfluorodecanoic acid (PFDA), perfluorobutane sulfonate (PFBS), perfluorohexane sulfonate (PFHxS) and perfluorooctane sulfonate (PFOS). Details on analytical method and quality controls are given in Part 1 (Boiteux et al. 2012).

Results and Discussion

The results obtained in the industrial park are presented in Table 1.

All the investigated monitoring wells of the industrial sites contained at least six perfluorinated compounds analyzed in this study, and the total PFC concentrations ranged from 2.08 µg/L (P01D) to 28.3 µg/L (P03A). Among the 10 PFCs analyzed, 6 PFCs (PFBA, PFPeA, PFHxA, PFHpA, PFOA and PFNA) were detected in every water sample. PFDA was also a prevalent PFC, but present at relatively lower concentrations. In contrast, the water samples from monitoring wells were generally free of perfluoroalkyl sulfonates (PFBS, PFHxS and PFOS).

Table 1 **PFC** concentrations in µg/L obtained from the FTP plant and the river

Location	P01A	P01D	P02A	P02D	P03A	NB	SB
PFBA	0.29	0.11	1.08	1.16	1.49	0.002	0.014
PFPeA	0.99	0.41	3.68	4.20	4.50	0.054	0.035
PFBS	n. q.	n. q.	n. q.	n. q.	n. q.	n. q.	n. q.
PFHxA	0.98	0.40	3.35	4.41	6.26	0.054	0.059
PFHpA	0.52	0.19	1.91	2.18	3.55	0.038	0.041
PFHxS	n. q.	0.02	n. q.	n. q.	0.13	0.005	n. q.
PFOA	0.72	0.63	3.19	2.77	9.14	0.067	0.103
PFNA	0.04	0.07	0.91	0.15	1.07	0.007	0.023
PFOS	n. q.	0.16	n. q.	n. q.	1.52	0.010	0.008
PFDA	n. q.	0.07	0.30	0.13	0.62	0.007	0.062
Total PFCs	3.54	2.08	14.4	15.0	28.3	0.24	0.34
Location	**WI 24 h**	**WI 7d**	**WE 24 h**	**WE 7d**	**RW1**	**RW2**	**RW3**
PFBA	0.46	0.67	2.22	2.11	<0.004	<0.004	<0.004
PFPeA	0.26	0.40	9.35	8.84	<0.004	0.004	0.004
PFBS	n. q.	n. q.	0.11	0.20	<0.004	<0.004	<0.004
PFHxA	2.50	3.11	13.3	8.67	<0.004	0.006	0.007
PFHpA	0.23	1.04	4.98	15.6	<0.004	0.005	0.006
PFHxS	n. q.	n. q.	n. q.	n. q.	<0.004	<0.004	<0.004
PFOA	0.64	1.66	9.08	15.1	<0.004	0.010	0.007
PFNA	n. q.	n. q.	0.74	2.89	<0.004	<0.004	<0.004
PFOS	n. q.	n. q.	n. q.	n. q.	0.006	0.005	0.006
PFDA	0.09	0.86	0.05	0.20	<0.004	<0.004	<0.004
Total PFCs	4.18	7.75	39.8	53.6	0.006	0.030	0.030

n. q. not quantifiable due to dilution or matrix effects

However, due to the high concentrations of some PFCs and to minimize matrix effects, the samples were highly diluted. Therefore, limits of quantification (LOQs) were increased. The monitoring well located near the river (P03A) was the most contaminated of the five investigated monitoring wells. PFOA (9.14 µg/L) and PFHxA (6.26 µg/L) were the predominant species. To summarize the results obtained in the monitoring wells, contamination was mainly due to perfluoroalkyl carboxylates and the two aquifers were equally affected by the industrial activity. An increase of the total PFC level was observed between P02A and P03A, in accordance with the groundwater flow direction.

Except PFHxS, all the monitored PFCs were present in runoff water, but the levels were lower than those reported from the monitoring wells (less than 0.1 µg/L). PFOA and PFHxA were again the predominant compounds, but PFDA showed a substantial concentration in SB. Surprisingly, both run-off basins (NB and SB) were contaminated, although the FTP plant is located in an area where run-off waters are only collected in SB. Atmospheric transport of PFCs from the FTP plant may explain their presence in both basins (Ruan et al. 2010).

Six PFCs were quantified in the FTP plant's effluents (WI). Only perfluoroalkyl carboxylates (PFBA, PFPeA, PFHxA, PFHpA, PFOA and PFDA) were found in the average weekly composite sample (WI 7d) and the average daily composite sample (WI 24 h). The total PFCs concentrations ranged from 4.18 µg/L (WI 24 h) to 7.75 µg/L (WI 7d), with PFHxA accounting for 40 %–60 % of total PFCs. The composition profiles of PFCs found in the FTP plant's effluents were slightly different. In the average daily composite sample (WI 24 h), the composition profile was PFHxA>PFOA>PFBA>PFPeA>PFHpA>PFDA, whereas it was PFHxA>PFOA>PFHpA>PFDA>PFBA>PFPeA in the average weekly composite sample (WI 7d). As WI 24 h was collected five weeks later than WI 7d, these differences suggest that the composition of the effluent may vary according to the manufactured products (fire-fighting foams, oil and water repellents) and the chemical processes used in the FTP plant.

Compared to the FTPs plant's effluents, a dramatic increase in PFC concentration was observed in WWTP effluents (WE 24 h and WE 7d). The total PFC concentration ranged from 39.8 µg/L (WE 24 h) to 53.6 µg/L (WE 7d). Eight PFCs were quantified, mostly perfluoroalkyl carboxylates. PFHxS and PFOS were not found in any of the samples. PFOA, PFHpA, PFPeA and PFHxA were the most predominant PFCs. Surprisingly, PFNA and PFBS were present in the end-stage effluent of the WWTP, although they were not detected in the FTP plant's effluents, which is one of the influents of the WWTP. The estimated total weekly mass of discharged PFCs was 8 g for the FTPs plant's effluent and 420 g for the end-stage effluent of the WWTP. This corresponds to a 50-fold increase between the two effluents. Two hypotheses may explain these observations. First, another—as yet unidentified—facility uses high amounts of PFCs and discharges its effluent into the WWTP. However, according to our preliminary investigations, the use of PFCs has not been reported by any other industrial facility whose the effluents enter this WWTP. Second, PFCs present in the FTP plant's effluents (fluorotelomers, such as alcohols, iodides and acrylates) are totally or partially degraded into PFCA during wastewater treatment. This degradation pathway has already been described (Schultz et al. 2006; Sinclair and Kannan 2006; Yu et al. 2009).

The WWTP effluent generates about 7,700 m^3 of waste effluent each week. This in turn corresponds to the release of nearly 20 kg of the 10 analyzed PFCs per year to the downstream water bodies.

The results obtained from the DWTPs are presented in Table 2.

At plant DWA, PFCs were found in only DWA-4 water samples. This alluvial well is not the closest to the river, but, of the five wells of the field, it is the one that is pumped at the highest flow rate. Other explanations of this contamination include differences in the local geological characteristics of the aquifer. Five perfluoroalkyl carboxylates were quantified. PFPeA, PFHxA, and PFHpA were the most prevalent PFCAs in this well and the total PFC concentration was 0.070 µg/L. The composition profile of PFCs found in treated water was similar to that found in the raw water of well DWA-4, but the concentrations of each PFCA were threefold lower than those in raw water. These similarities suggest that the water treatment (sand filtration + chlorination) has no effect on the removal of PFCs, as previously described (Takagi et al. 2011). The decrease in the total PFC concentration observed in treated water was mainly due to dilution by water from the uncontaminated wells.

Surprisingly, the total PFC level of treated water from DWB (0.024 µg/L) was about 2.5-fold higher than raw water (0.009 µg/L). Only three PFCAs were quantified in treated water (PFHxA, PFPeA, and PFHpA), none of which were present in raw water. In contrast, PFOS and PFOA were detected at low levels in raw water and were not present in treated water. Water samples collected at the outlet of the two treatment processes provide an explanation. The removal rate of the membrane filtration treatment (DWB-MF) is 100 % with respect to the 10 analyzed PFCs and the LOQ of the analytical method used. High levels of PFCs ranging from 0.004 to 0.1 µg/L were found in the membrane filtration brine, confirming the removal efficiency of this stage of the drinking water treatment. At the outlet of the biological treatment (DWB-BT), the total PFC

Table 2 PFC concentrations in µg/L observed at the DWTPs

Location	DWA-1	DWA-2	DWA-3	DWA-4	DWA-5	Treated water
PFBA	<0.004	<0.004	<0.004	0.007	<0.004	0.004
PFPeA	<0.004	<0.004	<0.004	0.023	<0.004	0.012
PFBS	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004
PFHxA	<0.004	<0.004	<0.004	0.021	<0.004	0.011
PFHpA	<0.004	<0.004	<0.004	0.013	<0.004	0.007
PFHxS	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004
PFOA	<0.004	<0.004	<0.004	0.006	<0.004	<0.004
PFNA	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004
PFOS	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004
PFDA	< 0.004	< 0.004	< 0.004	<0.004	< 0.004	<0.004
Total PFCs	<0.004	<0.004	<0.004	0.070	<0.004	0.034

Location	DWB	DWB-MF	DWB-BT	Treated water
PFBA	<0.004	<0.004	0.009	<0.004
PFPeA	<0.004	<0.004	0.026	0.009
PFBS	<0.004	<0.004	<0.004	<0.004
PFHxA	<0.004	<0.004	0.031	0.010
PFHpA	<0.004	<0.004	0.019	0.005
PFHxS	<0.004	<0.004	<0.004	<0.004
PFOA	0.004	<0.004	0.006	<0.004
PFNA	<0.004	<0.004	<0.004	<0.004
PFOS	0.005	<0.004	0.006	<0.004
PFDA	<0.004	< 0.004	<0.004	<0.004
Total PFCs	0.009	<0.004	0.097	0.024

DWA-1 raw water of *DWA*-well number 1, *DWB* raw water of *DWB, DWB-MF* water after membrane filtration, DWB-BT water after biological treatment

concentration was 0.097 µg/L. PFOA and PFOS present in raw water were not removed by this treatment. Furthermore, four PFCAs not initially detected in raw water were observed at substantial levels. The PFC levels in the treated water results from the dilution of the water from the biological treatment (20 %) by the water from the membrane filtration process (80 %). These results suggest that precursors of PFCAs are always present in raw water. They are likely degraded during the biological treatment technique, as previously observed in the WWTP of the FTP plant.

Upstream from the FTP plant, only PFOS was present in the river sample (RW1) (see Table 1). Downstream from the FTP plant, four PFCAs and PFOS were found in the river water (RW2). These four PFCAs (PFHxA, PFOA, PFPeA, and PFHpA) were exactly the same PFCAs that were predominant in the industrial WWTP effluents (WE 7d and WE 24 h). Concentrations were lower due to dilution by river waters. The PFCs (PFBS, PFBA, PFNA, and PFDA) that were quantified in the industrial WWTP effluents at low levels (<5 µg/L) were not detected in the river sample for the same reason. At the foot of the DWA (RW3), the concentrations and the PFCs present were similar to those observed downstream from the FTP plant. In contrast, levels of PFHxA, PFPeA and PFHpA were higher in the alluvial well DWA-4. This increase in concentration after passage through the alluvial sediment may be due to the degradation of PFCA precursors. The PFBA occurrence in DWA-4 may also be due to degradation of precursors during aquifer infiltration. Because of the length of its fluorocarbon chain and the nature of its functional group, PFOS may be retained in alluvial sediment (Higgins and Luthy 2006). This behavior may explain why PFOS was not detected in DWA-4.

To conclude, this study indicates that this industrial facility has contaminated the local underground aquifers. In the industrial WWTP, an overall increase in PFCA mass flows was observed, most likely resulting from the degradation of precursors during the wastewater treatment. However, observations of high levels of PFCAs in an alluvial well and at the outlet of a biological treatment process suggest that some precursors are not degraded and are always present in the effluents. The occurrence of PFCAs in drinking water may result from the degradation of precursors (mainly fluorotelomers). This degradation process may occur during sediment infiltration or during the treatment of drinking water. In the present study, short-chain PFCs (PFBA, PFHxA, PFHpA, and PFPeA) were the main compounds generated by degradation processes. These breakdown products are known for their higher environmental mobilities (Higgins and Luthy 2006) and their resistance to removal by the common treatment techniques (Wilhelm et al. 2010). In regard to PFOA and PFOS, concentrations observed in drinking water were not greater than the health-based drinking water concentrations protective for lifetime exposure that have been defined for other countries. The lowest published guide value for PFOA in drinking water is 40 ng/L (Post et al. 2009). Provisional health-related indication values for other PFCs have also been proposed (Wilhelm et al. 2010). Based on these data and the ten analyzed PFCs, the drinking water supplied by DWA and DWB can be considered safe.

As the above conclusions are based on a single sampling campaign, extensive sample collection is required to improve knowledge on the behavior of PFC precursors in water treatment plants and aquatic ecosystems. Further research is needed to verify that precursors are not present in drinking water and that chlorination/ozonation of these compounds does not lead to the production of other perfluorinated disinfection by-products other than short-chain PFCAs.

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